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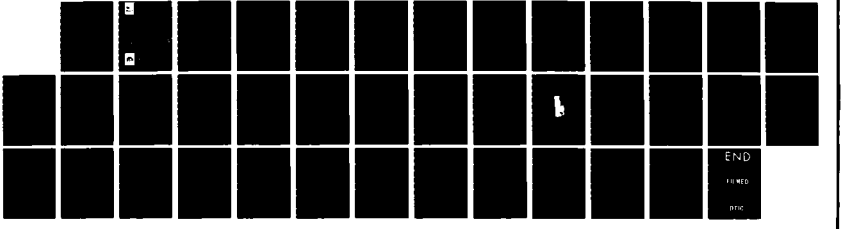

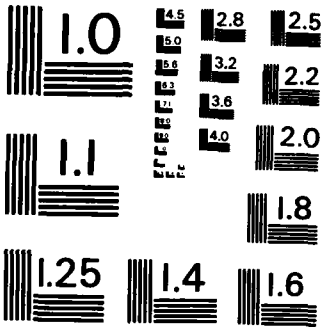

US Army Corps of Engineers

STRUCTURES LABORATORY

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DEVELOPMENT OF A SANDED EXPANSIVE SALT GROUT FOR REPOSITORY SEALING APPLICATION

by

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A sanded version of the basic BCT 1-F expansive salt grout was developed by the US Army Engineer Waterways Experiment Station in early 1983 for use in a test by Terra Tek in the Fall of 1983 in Salt Lake City. This grout is known as TT83.

Initial laboratory tests showed this mixture had adequate workability, impermeability, strength, and positive volume change.

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20. ABSTRACT (Continued).

There were problems with flow when this material was batched for the Terra Tek test. Later investigation indicated this was probably due to use of different equipment with lower mixing capacity.

Specimens made from this grout show over 0.1 percent expansion at an age between 56 and 90 days and about 0.3 to 0.4 at 1 year; expansion testing is being continued at 6-month intervals.

It was concluded that this mixture is probably satisfactory for use in repository sealing applications in a salt rock horizon.

Preface

This report was prepared for the US Department of Energy (DOE) under continuing contract DE-AI97-81 ET 46633. It was part of the final FY 84 milestone under that contract. Mr. Steve Webster of the DOE in Columbus, Ohio, was Project Manager.

The report was prepared in the Concrete Technology Division (CTD) of the Structures Laboratory (SL) of the USAE Waterways Experiment Station (WES) under the direction of Mr. J. M. Scanlon, Chief, CTD, and Mr. B. Mather, Chief, SL, by Messrs. A. D. Buck, J. A. Boa, Jr., and D. M. Walley. Mr. Buck was Project Leader.

COL Robert C. Lee, CE, was Commander and Director of WES during the conduct of the study. COL Allen F. Grum, USA, was Director of WES during the preparation and publication of this report. Mr. Fred R. Brown and Dr. Robert W. Whalin were Technical Directors.

Contents

Conversion Factors, Non-SI to SI (Metric)

Units of Measurement

Non-SI units of measurement used in this report can be converted to SI (metric) units as follows:

Multiply	By	To Obtain
cubic feet	0.02831685	cubic metres
inches	25.4	millimetres
Fahrenheit degrees	5/9	Celsius degrees or Kelvins*
feet	0.3048	metres
miles (US statute)	1.609347	kilometres
pounds (force) per square inch	6.894757	kilopascals
pounds (mass) per cubic foot	16.01846	kilograms per cubic metre

* To obtain Celsius (C) temperature readings from Fahrenheit (F) readings, use the following formula: $C = (5/9)(F - 32)$. To obtain Kelvin (K) readings, use: $K = (5/9)(F - 32) + 273.15$.

DEVELOPMENT OF A SANDED EXPANSIVE SALT GROUT FOR REPOSITORY SEALING APPLICATION

Background

1. A sanded version of salt grout BCT 1-F was developed in the Concrete Technology Division (CTD) of the Structures Laboratory (SL) of the US Army Engineer Waterways Experiment Station (WES) in 1983 for use by Terra Tek (TT) in its test with rock salt (halite) during September 1983. This grout mixture is now known as Terra Tek 83 (TT83).

2. Additional 1983 testing plus some testing started in June 1984 on this grout and variations of it are also reported.

3. In addition, the original CTD report on this grout dated 29 August 1983, subject, "Development of a Suitable Salt Grout for the 1983 Terra Tek Test," plus a CTD report dated 20 April 1984, subject "Additional Data on TT83 Grout," are included along with letters of transmittal and enclosures in order to have all of this together in one report (Appendices A, B, C, D).

Samples

4. Since no new materials were introduced into the grout, those identified in the first report (Appendix D) are unchanged with the possible exception that additional materials may have been obtained from the same sources. Such action is not considered significant.

5. Ten rock salt cores were obtained from Avery Island, Louisiana, on 29 March 1983 for possible use with this grout. Each core was 8 in. in diameter and about 16 in. long. They were given SL serial No. CL-43 DC-1 through 10.

Test Procedure

6. Additional expansion data and compressive strength data for the TT83 grout batched in the CTD on 13 July 1983 and 15 August 1983 were obtained.

7. Permeability tests were made as follows:

   a. Repeated tests were attempted on 6- by 6-in. salt cores cut from the larger salt cores from Avery Island, Louisiana. This was done

following the procedure of CRD-C 48-73* except that saturated salt water (37.2 percent salt) was used as the permeating medium. This test requires sealing of the bottom rim of the specimen on plaster and sealing the sides using paraffin-rosin and asphalt, but asphalt is not allowed to contact either the specimen, the container, or the permeating fluid. Driving pressure to move fluid through the specimen is 200 psi. All of these tests showed large water flow. The large water flow was found to be caused by conditions other than leakage due to improper sealing of the specimen in the apparatus. It was subsequently speculated that the flow was greater through the permeable pore space in the specimen than it would have through the same medium in situ in the earth because of the likelihood that an increased cross-sectional area of permeable pore space was created in the specimen after extraction due to spontaneous strain relief. Observations of specimens tested using brine containing a dye revealed brine movement through much of the intergranular spaces between salt crystals. In all cases the "large" flow was beyond the range for which the apparatus is intended to be used. Hence, if one desires to have a measure of the permeability of the rock in the state of restraint it possesses in the subsurface, one must maintain or restore such restraint before measuring permeability. The opportunity to do this when more than 200 psi is required is not provided by CRD-C 48-73. Therefore, a smaller 3- by 6-in. core was prepared and tested in a Hassler cell. This method uses higher confining pressure for sealing and has the ability to employ a range of confining pressures. The confining pressure involves fresh water under pressure acting on the specimen through a membrane. This salt core was tested at 200-psi driving pressure and 400-psi confining pressure. This specimen and other salt specimens described later were heated at about 115° F so the warm specimen would have less tendency to take moisture from the air during preparation for testing.

b. A 6- by 12-in. cylinder of the original unsanded BCT 1-F salt grout was cast in June 1983 and tested by CRD-C 48-73 as two 6- by 6-in. cylinders when it was about 90 days old.

c. A 6- by 12-in. cylinder of TT83 sanded salt grout was cast as part of the 13 July 1983 laboratory batch and tested as above when it was about 60 days old.

d. Some of the rock salt was cored and cut into 3- by 6-in. cylinders. These were cut down their long axis to make half cylinders. Each heated half cylinder was placed in a 3- by 6-in. cardboard mold that had been reinforced by wrapping with electrician's tape. One of these cardboard molds was filled with unsanded BCT 1-F salt grout on 12 July 1983 to create a simulated borehole (SBH) sample, containing a grout-rock interface, with the intention of testing it for permeability. The plan was to demold such a specimen when the grout hardened after about 48 hr and immediately place the specimen in a Hassler cell where confining pressure could be

* USAE Waterways Experiment Station, CE, "Handbook for Concrete and Cement," with quarterly supplements, Vicksburg, Miss., August 1949.

applied until permeability testing was started when a specimen was about 28 days old. The confining pressure was to be applied with fresh water acting through a membrane while driving pressure of 200 psi was to be with saturated salt water (i.e., 37.2 percent salt by weight of water). The first SBH sample was made before the TT83 sanded grout formulation was available so unsanded BCT 1-F salt grout was used. This SBH sample was placed in a Hassler cell under 300-psi confining pressure when it was 2 days old and left in that condition until permeability testing at 200-psi driving pressure started when it was 29 days old. Another SBH sample, this one made with TT83 sanded grout, was cast on 2 August 1983 and placed in a Hassler cell when it was 6 days old. However, an error was made in assembling the apparatus and the confining fresh water leaked through to the specimen and ruined it. The specimen was photographed both to show the general appearance of such a SBH sample and to show the effect of this leakage on the salt.

8. The chemistry of possible reactions between grout and salt at their contact surface was studied by X-ray diffraction (XRD) examination of salt surfaces before and after contact with grout plus some XRD examination of the grout. The intent was that chemical changes would be seen as changes in phase compositions. Accordingly, in May 1983, 18 small specimens of rock salt cut from Avery Island salt core CL-43 DC-1 were prepared (slabs 1S through 19S, No. 16S discarded), examined by XRD, and combined with unsanded BCT 1-F salt grout in small sealed plastic containers. Excess salt water was added to the hardened specimens before sealing. Eight of these SBH samples (CL-43 GRT-1-A through 1-H) were placed in storage at 60° C when they were 2 days old. The other 10 SBH samples (CL-43 GRT-1-I through 1-O and 1-Q, 1-R, 1-S) were kept at ambient laboratory temperature. The SBH samples were split open along the salt to grout contact as nearly as possible at 7-, 28-, 90-, 180-, and 365-day ages and examined by XRD.

9. The third sample of TT83 grout that had been dry batched at the CTD in September 1983, sent to Terra Tek in Salt Lake City, and later returned to the CTD was combined with water on 23 March 1984. The flow was checked; the material was then cast into 3- by 6-in. cylinders, 1- by 1- by 10-in. (effective gage length) bars, and 2-in. cubes. All of these were sent to Dr. P. Licastro at the Pennsylvania State University (PSU) for testing there (Appendix C). No specimens were retained at WES.

10. Six batches of salt grout were made to evaluate the effect of changes in composition on the sanded TT83 salt grout that had been developed in 1983. They included the following:

a. Mixture 1. This was a repeat of TT83 using some old and some new material from the original sources made on 5 June 1984. For this batch, tests of the unhardened mixture included 2-hr workability, air content, bleeding, density, and temperature of the grout. Twelve expansion bars, each 2- by 2-in. with 10-in. gage length, were cast. Three of these were restrained bars essentially as described in CRD-C 225-76 (ASTM C 806-75) and the rest were unrestrained. Use of restrained bars is the standard way to measure this type of expansion. The restraint against expansion is provided by a metal bar cast into the specimens and threaded into metal end plates. The three restrained bars were kept in moist storage at about 73° F. The nine other bars were covered until they were demolded and then sealed in plastic with tape. Three of these were kept in the moist room and three in a sealed plastic container in laboratory air at about 73° F while the final three were stored over water at 86° F. Readings were scheduled as for the strength tests. When it was observed that there were precipitates in the limewater that mixture 1 cubes were stored in plus brown flakes on cube surfaces, these materials were sampled and examined by XRD.

b. Mixtures 9, 10, 12, 14, and 27. These five were smaller batches of modified TT83 grout cast 7 June 1984; the differences in these will be described later. Due to small size of the batch, fewer properties of the unhardened grouts could be determined than for mixture 1. Those that were determined included 2-hr slump, density, and temperature of the grout. Cubes were cast for compressive strength determinations at 7, 14, 21, 28, 56, 90, 180, and 365 days. The cubes for mixture 12 were discarded when they failed to harden all the way from top to bottom 5 days after they had been made.

11. All XRD patterns were made with an X-ray diffractometer using nickel-filtered copper radiation.

12. A batch of TT83 grout was cast 6 September 1984; most of it was placed in several instrumented pipes for monitoring of expansion and stress levels; expansion bars were also cast for periodic readings. The results of this work will be reported separately.

Results

13. Table 1 in Appendix D shows the mixture proportions for this grout. Tables 2 and 3 in Appendix D for TT83 grout were revised to include more data. Table 2 shows expansion is still increasing at 1-1/2 years and is now about 0.4 percent for restrained bars kept in a favorable environment (approximately 73° F and 100 percent R.H.). These measurements will be continued at 6-month intervals. The only change in Table 3 was addition of 28-day compressive

strength values for TT83 cubes cast 15 August 1983. Twenty-eight-day strength was above 5000 psi for two slightly different formulations of this grout. All of the cubes have now been broken.

14. Table 4 shows the results of permeability testing. As already mentioned, this testing of salt cores by the permeability test method used for concrete (CRD-C 48-73) was a consistent failure. When a 3- by 6-in. salt core was tested in a Hassler cell using a confining pressure of 400 psi, the permeability was about 100 microdarcies after 2 days of testing. While this was low permeability than was indicated in the C 48 tests, it is likely that more confining pressure would have resulted in still lower permeability.

15. The permeability value for the unsanded BCT 1-F salt grout was 2 microdarcies; the fact that the seal around the other 6- by 6-in. specimen leaked is quite common since the encapsulation procedure used results in some failures. The sanded TT83 salt grout showed no detectable permeability after testing two 6- by 6-in. cylinders for 21 days. These extremely low values for these grouts are usual since water to cementitious solids ratios have been reduced to 0.3.

16. The SBH sample that had a contact surface of salt and unsanded BCT 1-F salt grout was satisfactory for 6 days of testing with a permeability of 2 microdarcies. However, after that the flow rate increased and subsequent inspection showed that the test solution had dissolved its way along the contact of salt and grout. The solution was made using 37.2 percent salt by weight of water dissolved. This is saturation with salt at atmospheric pressure. It is now believed that increased solubility of salt at increased pressure in the range used permitted some dissolution of salt which then led to increased permeability. This would also have been true for the salt core tested by itself. Future testing will need to recognize the fact of increased salt solubility with increased pressure and be modified to accommodate this without effect on permeability data. Photograph 1 shows the general appearance of these SBH samples and shows the effect pure water had on this sample that could not be teste because of this attack.

17. Table 5 show expansion data for a batch of sanded TT83 grout cast 5 June 1984; this is the same grout as that cast 13 July 1983. Comparison of 90-day data for bars kept at about 73° F and 100 percent R.H. shows good agree ment (approximately 0.1+ percent). Table 5 also includes data for bars from the same grout that were sealed and stored at about 73° F and 86° F. These

results show the same sort of results as similar testing with sanded nonsalt grout TT84.* These results indicate lack of effective sealing leading to drying with shrinkage or reduced expansion and less expansion at the higher temperature.

18. Table 6 shows data for freshly-mixed grouts, compressive strengths, and major variables for TT83 grout (mixture 1) and modifications of it (mixtures 9, 10, 12, 14, 27). Comparison of 7- and 28-day strengths for TT83 grout in this table and in Table 3 show satisfactory agreement. Mixture 12 cubes were discarded since they failed to harden after 5 days, and this was regarded as too long to be practical. Mixture 14 has strengths that are lower and only marginally acceptable; mixtures 9, 10, and 27 have more than adequate strength. These variations on TT83 will be discussed later.

19. There was not time for detailed study of the XRD results of examining salt surfaces before and after contact with unsanded BCT 1-F grout as SBH samples. Since there will be at least one more report to include continued expansion testing of TT83 grout cast 13 July 1983, 5 June 1984, and 6 September 1984 plus more strength data for the June grout, the XRD data will be deferred until that next report.

20. As indicated earlier, the flow time for dry batched TT83 grout returned from Terra Tek was satisfactory when it was mixed with water in the CTD on 23 March 1984. All of the specimens that were made were sent to PSU and any reporting of them will be by PSU.

21. Examination of material precipitated in limewater during storage of TT83 cubes and of material on cube surfaces by XRD showed:

a. The precipitates were halite (NaCl), sylvite (KCl), and thenardite (Na_2SO_4) plus calcite ($CaCO_3$) or aphthitalite ($(K,Na)_2SO_4$) or a complex alkali chloride sulfate compound. These are probably normal compounds to form from storage of these grout cubes in limewater.

b. The brownish material scraped from cube surfaces was a combination of calcite with some quartz and halite. These materials too are considered normal.

Discussion

22. A major intent of additional work on TT83 grout this FY was to investigate the effect of changes in composition on the grout. Review of this

* Buck, A. D., "Development of a Sanded, Nonsalt, Grout (TT84) for the 1984 Terra Tek Test," USAE Waterways Experiment Station Grout Report dated 12 July 1984.

plan showed that changes in amount of cement, in amount of fly ash, in amount of plaster, and in amounts of high-range water reducer (HRWR), water, and aggregate had already been evaluated in the FY 83 development of the TT83 grout. The amounts recommended for TT83 had evolved from those considerations. Since changes in the basic materials of cement, fly ash, plaster, and aggregate had already been investigated, the changes embodied in mixtures 9, 10, 12, and 14 (Table 6) were intended to investigate the effects of deletions or substitutions that amounted to fairly minor changes. The changes from the basic TT83 grout that are represented by mixtures 9, 10, 12, 14, and also by 27 are listed below:

a. Mixture 9. Omission of De-Air additive.

b. Mixture 10. As above plus used a naphthalene type HRWR for the melamine type.

c. Mixture 12. No De-Air plus a liquid melamine type HRWR for the powder melamine type.

d. Mixture 14. Use of the naphthalene type HRWR with De-Air for comparison with mixture 10.

e. Mixture 27. Use of local natural siliceous sand for the three siliceous fine aggregates usually used (20-40 sand, D-30 sand, and silica flour).

23. The strength data in Table 6 indicate that use of the liquid type melamine HRWR in mixture 12 probably resulted in excessive delay of set time since lack of De-Air material probably had no effect on this property of the grout. The only other significant effect shown in Table 6 is the lower strength of mixtures 10 and 14; this was probably due in each case to higher air contents associated with use of the naphthalene type HRWR. Although air was not measured for mixtures 9, 10, 12, 14, and 27 due to small batch size, the increased flow times of mixtures 10 and 14 suggest higher air contents due to the foaming associated with this naphthalene type HRWR. The same effect seems indicated for mixture 12 that was discarded due to failure to set. Aside from lower strengths and the tendency of the coarser sand in mixture 27 to fall out, other changes were not significant in the five modified grout mixtures.

Conclusions

24. Based on data from the original and second reports (Appendices D, A) plus new results, the following conclusions appear reasonable.

a. The sanded salt grout known as TT83 is a satisfactory repository sealing mixture for use in salt host rock. This is based on the

fact that it has extremely low permeability (<5 microdarcies), compressive strength above 5000 psi by its 28-day age, and expansion in a favorable environment (approximately 73° F, 100 percent R.H.) of about 0.1 percent by 90 days and about 0.4 percent by its 365-day age. In addition, plastic properties are satisfactory. They include workability for at least 2 hr with flow of about 20 sec, and less than 1 percent bleeding.

b. While the exact cause of excessive flow time encountered for the Terra Tek test in September 1983 has not been specifically identified, it seems likely this was due to batching with inappropriate or less appropriate equipment than used in the CTD (Appendix D). The slight addition of water that was made to that grout shortly after it was mixed is not believed to have caused any significant lowering of important properties.

c. While efforts to determine expansion under other than ideal conditions were attempted, these generally failed because of inability to consistently obtain effective sealing. As with work on the nonsalt TT84 sanded grout,* the general indication is inhibition of expansion with sealing and elevation of temperature.

d. The use of fresh supplies of some materials from the original sources did not have any significant effect on a repeat batch of TT83 grout.

e. Earlier changes in amounts of materials or in deletion or replacement of some materials with other materials for the TT83 grout have indicated that the present formulation is about optimum for the properties that have been monitored. The form and type of HRWR used does have some impact and should not be changed without due consideration.

Recommendations

25. Since expansion testing of TT83 grout is continuing beyond 1 year, it is recommended that these data be prepared in one or more subsequent reports to be issued at later dates. This report should include unreported XRD data on study of salt surfaces before and after contact with grout similar to TT83.

26. Testing of SBH samples representing contact of salt and TT83 grout to determine permeability at this contact surface should be pursued.

27. Testing of TT83 grout batched and cast into metal pipes on 6 September 1984 to monitor expansion and stress levels should be continued as should testing of companion specimens cast at the same time.

Table 1

Mixture Data for Sanded Modification of BCT-1-F Salt Grout

Constituents	Amount Required for 1-ft^3 Batch, lb
RC-681 Class H Cement*	39.32
AD-592(2) Fly Ash*	13.21
Cal-Seal (Plaster)*	4.63
Melgran 0**	0.86
Salt (NaCl)†	7.69
De-Air No. 1	0.1422
20-40 Sand	20.25
D-30 Sand	20.25
Silica Flour	8.11
Water	20.69
Ratio of Water to Cementitious Materials (0.36)	
Flow was 21 sec at 5 min, 20 sec at 60 min, and 19 sec at 120 min	
Bleeding was 0.18 percent	
Actual Unit Weight, 136.43 lb/ft^3	

* Considered to be cementitious materials.

** Melamine powder high-range water reducer (superplasticizer); 1.50 percent used by weight of cementitious materials.

† 37.2 percent by weight of water (BWOW).

Table 2

Restrained Expansion of BCT 1-F Salt Grout Containing Sand[a]

Age, days	Length Changes, %: 3- by 3- by 10-in. Bars No. 12	No. 25	Average	2- by 2- by 10-in. Bars No. 43	No. 47	Average
5	0.013	-0.010	0.002	0.001	0.006	0.004
7	0.030	0.031	0.030	0.011	0.015	0.013
28	0.056	0.061	0.058	0.044	0.031	0.038
47	0.073	0.082	0.078	0.059	0.051	0.055
90[b]				0.129	0.093	0.111
180	0.184	0.210	0.197	0.218	0.191	0.204
365	0.353	0.384	0.368	0.387	0.368	0.378
Age, years						
1-1/2	0.475	0.409	0.442	0.471	0.459	0.465

(a) Cast 13 July 1983; stored and measured at 23 ± 1.7° C. Since bars were not stripped until they were 5 days old, the reference length is the cage length before casting.

(b) Data for bars 12 and 25 were in error and were deleted.

Table 3

Compressive Strength of Two Modifications of a Sanded Salt Grout

Mixture	Individual Cubes	Compressive Strength (psi) of 2- by 2- by 2-in. Cubes at Ages Shown Below, days			
		3	5	7	28
Cast 13 July 1983[a]		Not tested	2000	2940	4360
			1950		5560
					5960
	Average		1980	2940	5290
Cast 15 August 1983[b]		260	Not tested	3360	5840
		210		3540	5800
				3480	5400
					5670
	Average	240		3460	5680

(a) Six 2-in. cubes were made and broken in groups of two, one, and three.

(b) This formulation was considered best because bleeding was less than the 13 July mixture and flows were about the same. Nine 2-in. cubes were made and broken in groups of two, three, and four cubes.

Table 4

Permeability Data for Rock Salt, for Unsanded and Sanded BCT 1-F Salt Grout, and for One Simulated Borehole (SBH) Sample

	Permeability, microdarcies
CRD-C 48-73 Method (6- by 6-in. cylinders, 200-psi driving and confining pressure, except on exposed bottom surface)	
1. Unsanded BCT 1-F salt grout, ~90 day age	~2 for one 6- by 6-in. cylinder, other leaked
2. Sanded BCT 1-F salt grout (TT83), ~60-day age	Zero after 21 days of testing two 6- by 6-in. cylinders
Hassler Cell Method	
3. Rock salt core, ~3- by 6-in. size, using 400-psi confining pressure and 200-psi driving pressure	~100 after 2 days
4. SBH 3- by 6-in. sample (salt unsanded BCT 1-F grout), ~29-days age, 300-psi confining pressure and 200-psi driving pressure	~2 after 6 days, the sample leaked

Table 5

Expansion Data for Sanded TT83 Salt Grout at Two Temperature and Moisture Conditions[a]

Age, days	Expansion, %, 2-in. Bar No. I	II	III	Average	
73° F, 100% R.H.[b]					
7	0.026	0.026	0.023	0.025	
14	0.044	0.041	0.037	0.041	
21	0.056	0.055	0.049	0.053	
28	0.074	0.073	0.061	0.069	
56	0.105	0.106	0.101	0.104	
90	0.142	0.146	0.143	0.144	
180	0.211	0.222	0.212	0.215	
365	0.283	0.290	0.283	0.285	
73° F, Sealed (Kept in moist room)	IV	V	VI		
7	-0.017	-0.010	-0.024	-0.017	
14	0.007	0.007	-0.005	0.003	
21	0.015	0.013	0.007	0.012	
28	0.028	0.018	0.013	0.020	
56	0.032	0.030	0.027	0.030	
90	0.050	0.037	0.039	0.042	
180	0.055	0.119	0.065	0.080	(0.060, 2)
365	0.097	0.122	0.137	0.119	
73° F, Sealed (Plastic container)	VII	VIII	IX		
7	-0.027	-0.025	-0.030	-0.027	
14	-0.042	-0.040	-0.041	-0.041	
21	-0.051	-0.049	-0.049	-0.050	
28	-0.055	-0.054	-0.054	-0.054	
56	-0.035[c]	-0.076	-0.073	-0.074	
90	-0.097	-0.100	-0.093	-0.097	
180	-0.123	-0.127	-0.115	-0.122	
365	-0.156	-0.159	-0.152	-0.156	
86° F, Sealed (Over water in plastic container)	X	XI	XII		
7	-0.026	-0.028	-0.027	-0.027	
14	-0.022	-0.021	-0.021	-0.021	
21	-0.002	-0.006	-0.004	-0.004	
28	0.005	0.005	0.005	0.005	
56	0.009	0.012	0.011	0.011	
90	0.025	0.033	0.027	0.028	
180	0.041	0.051	0.035	0.042	
365	0.156	0.064	0.055	0.092	

(a) Cast 5 June 1984.
(b) Bars I, II, III are restrained; all others are not.
(c) Value not used for calculation of average.

Table 6

Compressive Strength of Sanded TT83 Salt Grout[a] and Modifications of It[b] at Different Ages

	Compressive Strength, psi, of Mixture Shown[c]					
Age, days	1	9	10	12	14	27[d]
7	2,780	2810	1980	Discarded	2060	3430
14	4,320	3920	2490	due to fail-	2120	4050
21	5,860	4760	3280	ure to hard-	2980	5370
28	7,280	5840	2000	en after 5	3120	6090
56	7,130	7290	4080	days	3650	7350
90	10,540	7680	5140		3120	8750
180	11,710	9910	5480		4850	No specimens left
Plastic Properties						
2-hr Flow, sec	20 to 23	26 to 24	26 to 32	31 to 36	32 to 24	16 to 15
Grout Temperature, °F	70 to 76	72[e]	72[e]	72[e]	70[e]	70[e]
Wet Density, lb/ft^3	137.6	132.2	115.6	118.9	133.1	137.6
% Air	0.2			Not determined		
Bleeding	Not available			Not determined		
Modification of TT83 Grout	None	Omit De-Air	Omit De-Air, use naphtha-lene HRWR	Omit De-Air, use liquid melamine HRWR	Use naphthalene HRWR with De-Air	Different aggregate

(a) Cast 5 June 1984 as mixture 1.
(b) Mixtures 9, 10, 12, 14, and 27 cast 7 June 1984.
(c) Values may be average of two or three 2-in. cubes or values for single cubes.
(d) Some fallout.
(e) Initial temperature only.

Photograph 1. View of ruined SBH sample made of TT83 grout (left) and rock salt (right) after fresh water reached it. The dissolution of salt along the contact surface is evident.

APPENDIX A

20 April 1984 Grout Report

DEPARTMENT OF THE ARMY
WATERWAYS EXPERIMENT STATION, CORPS OF ENGINEERS
P.O. BOX 631
VICKSBURG, MISSISSIPPI 39180

REPLY TO ATTENTION OF

April 23, 1984

Structures Laboratory

Dr. Roger Wu
Project Engineer, Salt Repository
Project Office
US-DOE Columbus
505 King Avenue
Columbus, Ohio 43201

Dear Dr. Wu:

The enclosed report deals with the problem of flow time for the grout (TT83) used for the Terra Tek test last September (Enclosure). This is not an FY 84 milestone report.

Sincerely,

A. D. Buck

A. D. Buck
Concrete Technology Division
Structures Laboratory

Enclosure

Copy furnished
(with enclosure):

Don Moak, ONWI
Dan Bush/Dick Lingle, Terra Tek
Della Roy, PSU
Dave Lankard, Consultant
Peter Kelsall, D'Appolonia

Corps of Engineers, USAE Waterways Experiment Station	Grout Report	Structures Laboratory P. O. Box 631 Vicksburg, Mississippi
Project Additional Data on TT83 Grout		Date 20 April 1984 ADB

1. During FY 83, a sanded version of salt grout BCT-1-F was developed by the Concrete Technology Division (CTD) of Waterways Experiment Station (WES) for use during a laboratory test at Terra Tek (TT) in September and October 1983. That grout has since become known as TT83 and will be referred to that way in this report.

2. This report discusses a problem with the TT83 grout that occurred when it was mixed with water at TT and subsequent developments. Pertinent items will be listed and then discussed.

a. The CTD provided a report dated 29 August 1983 concerning development of this grout by letter to Don Moak dated 1 September 1983 (Enclosure 1) The letter also stated that dry-batched samples of this grout plus some extra cement would be sent to TT that same week. This was done.

b. One of these dry batches had been mixed (water added) by TT prior to the test date. On 20 September, when the actual batch to be used in the test was mixed, it was found to have a flow time far in excess of the 19 to 21 sec recorded in Enclosure 1. As an expedient measure, John Boa of the CTD added a little water to the mixture to reduce flow time. After this was done, the placement continued. The extra water increased the ratio of water to cementitious solids (cement, fly ash, plaster) from 0.36 to 0.39. Pennsylvania State University (PSU) data on hardened samples of this particular grout mixture have been generally satisfactory.

c. At that point the CTD was to have no more direct contact with the test or any posttest examination of the grout. John Boa expressed the hypothesis during a meeting at ONWI in October 1983 that the excessive flow time found upon initial mixing of TT83 may have been due to a batching error at WES such that the amount of high-range water reducer (HRWR, superplasticizer) was too low. Later during discussions between Dan Bush of TT and PSU, there was concern that the total weight of the unused third batch was too high. Therefore, this batch was returned to WES. At that time, John Boa examined this batch and weighed the different portions. This inspection did not support his earlier thought that the amount of HRWR might be low. In addition, the weights showed that if the 20 or so lb of extra cement that had been sent was accounted for, then the weights were as they should be. This was extra cement for another purpose and was not and should not be included in the calculations. It was not labeled as being part of any batch.

d. On 23 March 1984, the third batch of TT83 grout that had been returned to the CTD was mixed by adding the prescribed amount of water and combining materials. Specimens were cast and most were later sent to PSU for their use as they had requested. This is described in a letter from Don Walley to Pat Licastro of PSU, dated 3 April 1984 (Enclosure 2). While this letter does not specifically make the point, the fact is that this batch

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Rev Feb 1970

Enclosure 1

of that grout was essentially like the developmental mixture of Enclosure 1 and did not have an excessive flow time.

e. On 3 February 1984, Dr. Judith Moody of ONWI forwarded to John Boa of CTD a copy of a letter from Dr. D. Roy, dated 31 January 1984, concerning the TT83 test. Dr. Moody's note asked John Boa to comment. He did this by letter dated 19 April 1984 (Enclosure 3).

2. The following comments seem appropriate in view of what is known:

a. There is no specific explanation for the excessive flow time of the TT83 grout batched last September. Since the third batch of the same grout behaved normally when mixed at CTD recently, it is believed that the problem was largely or wholley due to the lack of proper mixing equipment to do all of the grout mixture at one time. This belief is supported by CTD experience through the years that grout needs to be mixed in a high-speed mixer that imparts a high shearing action. As an aside, the proper type of mixing is not obtained with a conventional concrete mixer having a much slower speed of rotation. Therefore, it is strongly recommended that TT be sure to have adequate mixing equipment for the next test with TT84 grout (i.e., high speed, high shearing action, adequate volume).

b. PSU has found in subsequent work with this mixture that the setting times they obtained were far longer than those found by WES. Discussions between PSU and WES (largely D. Walley) about TT83 grout setting time have indicated that the difference PSU found was largely due to the fact that CTD stored fresh specimens at about 23° C (73° F) while PSU used an elevated temperature of 38° C (100° F). In addition to effect of temperature on setting time, this emphasizes the fact that the expansion of this or any expansive mixture is likely to be sensitive to temperature since the basic requirement is for proper timing so that expansion occurs when the material has set and has the proper strength. If expansion is too soon, it may occur in a plastic rather than a rigid material and be lost. If expansion is too late, much of it may be lost because the rigid material has too much strength. Therefore, temperature effect on setting time and on expansion will receive attention for further study of the TT83 sanded salt grout and for the development of a sanded nonsalt grout this FY.

3. These statements are in general agreement, where there is overlap, with conclusions reached independently by PSU.

APPENDIX B

Letter from Boa to Moody

DEPARTMENT OF THE ARMY
WATERWAYS EXPERIMENT STATION, CORPS OF ENGINEERS
P.O. BOX 631
VICKSBURG, MISSISSIPPI 39180

April 19, 1984

REPLY TO ATTENTION OF

Structures Laboratory

Dr. Judith Moody
Office of Nuclear Waste Isolation
505 King Avenue
Columbus, Ohio 43201

Dear Judith:

This is in response to the letter you forwarded to me from Della, dated January 31, 1984, concerning the Terra Tek September 3 grout placement.

A recap of what occurred at Terra Tek is that the mixture used did not behave in the fluid state as it should have. I do not know why. Hindsight says I should have discarded the mixture and used the third batch that was there, because when we put the third batch together here at WES in March 1984, after it was shipped back from Terra Tek, it behaved properly. I increased the water slightly, from a designed water/cement ratio (by weight) of 0.36 to 0.39 (i.e., water/cement plus fly ash plus cal seal) in an attempt to decrease the flow. This was moderately successful; however, the flow did not approach what we had gotten in the laboratory. The mixture was placeable, and I have confidence in the resulting homogeneity of the batch.

The third batch, shipped back to WES by Terra Tek, was put together in March 1984 with a flow of 24 seconds. Specimens specified by Pat Licastro were cast for future shipment to PSU. In addition, we cast nine 4- by 8-in. cylinders and one 12-in. cylinder to keep at WES. It is suggested that PSU compare the Terra Tek September 1983 test results with the specimens supplied in 1984 from the third batch.

In response to specific comments in the PSU letter, I offer the following:

Page 3, first table. The gray powder is cement. It was supplied to be used to seal leaks, if any occurred, at Terra Tek. It is not part of the mixture and was not marked as part of Batch 3. The other ingredients in the table are essentially correct or as close as they could be weighed in the plastic bags.

Page 3, second table. Same basic comments as for first table.

Page 3, third table. Based on comments for tables 1 and 2, if you subtract 16.27 lb from 130.89 lb, you get 114.62 lb, which is very close to the correct weight.

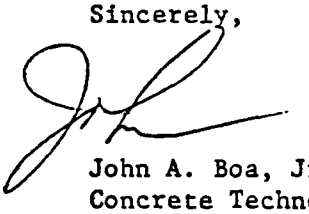
Page 2, table. The cement weight should be 39.32, not 39.22.

I have no comments on the reported greater than 14-day set time experienced by PSU. All the mixtures we have proportioned at WES have set in approximately three days including the one from Batch 3 recently mixed.

I have enclosed a copy of pages 2 and 3 of PSU letter with comments in the margins (Enclosure 1). I think this will clear up some of the discrepancies. Note specifically the weight comparison from Table 2, page 3, and that on page 2 (i.e., 105.93 lb vs. 105.77 lb). These are close enough to be called identical. The weights of Batch 3, returned from Terra Tek to WES, checked out okay.

I hope this lays the problem to rest. I'm sorry it happened. We will guard against this reoccurring in the future.

Sincerely,



John A. Boa, Jr.
Concrete Technology Division
Structures Laboratory

Enclosure

permeability flow test that there appeared to be a discrepancy between the stiffness of the mix made at the time of the test and that obtained at WES. This was discussed in some length at the meeting held at Columbus, October 1983.

The remedial action taken on site consisted of adding one litre of brine (lacking 10.6 oz of salt of being saturated [reference, Lankard 9/30/83 Trip Report]). This decreased the flow from 120 seconds to 70 seconds, but it was still higher than the 20 seconds measured at WES. The decision to continue was made and the test specimen cast, along with the parallel test samples for MRL/PSU. The equivalent formulation based upon the notes of Dave Lankard (20 Sept 83) is reflected below:

	TTS83-1 As proposed	TTS83-2 Lankard
cement	39.22	39.22
fly ash	13.21	13.21
plaster	4.63	4.63
superplasticizer	0.86	0.86
salt	7.69	7.03
de-air	0.1422	0.1422
sand (80-40 + D-30)	40.5	40.5
silica flour	8.11	8.11
water	20.69	22.89
Total mass (lbs)	135.05	136.59

This still leaves the question as to the reason for the discrepancy in the flow behavior which could have a potential impact upon the interpretation of the parallel and post-test studies. A resolution to this impasse appeared possible as WES had provided three identical batches, each packaged independently, to Terra Tek. The third remaining batch could be opened and the component weights determined. This may allow the identification of a discrepancy in the preparation of the dry batch grout formulation. With the consent of Donald Moak, Dan Bush at Terra Tek was requested to open and weigh the remaining untouched grout batch.

The following observations were made at Terra Tek by Dan Bush upon opening the third box of grout ingredients. The box contained four unmarked plastic bags, one of which contained three plastic bottles. One bag contained a fine gray powder tentatively identified as cement, another bag contained a light gray to buff colored, fine powder that was phase separating into a white component (perhaps the fly ash and plaster and/or silica flour), another bag contained the sand and the fourth bag contained the salt. Two of the three small plastic bottles were filled and the third bottle was only partially filled. The following masses were recorded for each of these components (telephone communication D. Bush to B. Scheetz):

Enclosure 1

		presumed content	
gray powder		16.4 lbs	(cement) ← EXTRA CEMENT NOT PART OF BATCH
light gray to buff powder	106.19	33.32 lbs	(fly ash +)
sand		72.87 lbs	(sand)
salt	OK	7.82 lbs	(salt)
powder bottle #1		0.51 lbs	
powder bottle #2 (similar to #1)	OK	0.50 lbs	(Melgram-O)
powder bottle #3		0.20 lbs	(de-air)
		131.62 lbs. gross	

The mass of the plastic bag was taken to be the difference between the observed mass of the salt, 7.82 pounds, and the reported 7.69 pounds or 2.08 ounces, a reasonable estimate. All of the masses were adjusted to account for this mass of the bag. In a similar manner, the mass of the apparent superplasticizer at 1.01 pounds was subtracted from the reported 0.86 pounds, halved and the mass of 1.2 ounces used for the mass of the plastic bottles. The formulation in the remaining batch, therefore, would be:

cement		16.27 lbs	← EXTRA CEMENT NOT PART OF BATCH
flyash + plaster + silica flour	105.92	33.19 lbs	
sand		72.74 lbs	
salt		7.69 lbs	- O/L
superplasticizer		0.86 lbs	- OK
de-air		0.14 lbs	- OK
total mass (lbs)		130.89 lbs net	

The mass of the solids determined by Terra Tek contrasts to the other two formulations accordingly:

Proposed	114.36 lbs
Lankard	113.70 lbs*
remaining batch	130.89 lbs (minus 16.27 = 114.62) - OK

*difference in mass due to removal of salt.

The three batches were reported to be identical, so that in use at Terra Tek, no specific order was followed in their utilization. Dan Bush reported in a telephone conversation that the box marked #2 was used in the 'dry run' mix, the box marked #3 was used in the actual test and the box marked #1 remained and was the one used to obtain the above masses of ingredients.

The results of the noted discrepancies in mass were reported to Donald Moak via a telephone conversation on the 9th of November 1983.

TTS-83-1 Setting Time Variations

In the process of making some post test analysis of the Terra Tek test, several trial batches of the proposed TTS-83-1 formulations were prepared

APPENDIX C

Letter from Walley to Licastro

DEPARTMENT OF THE ARMY
WATERWAYS EXPERIMENT STATION, CORPS OF ENGINEERS
P.O. BOX 631
VICKSBURG, MISSISSIPPI 39180

April 3, 1984

REPLY TO
ATTENTION OF

Structures Laboratory

Mr. Pat LiCastro
103 Materials Research Laboratory
The Pennsylvania State University
University Park, Pennsylvania 16802

Dear Pat:

Here are the specimens you requested be cast from the Terra Tek 83 salt-sanded grout mixture returned to WES from Terra Tek by John Boa.

One cubic foot of the mixture was mixed on Friday, March 23, and the following specimens were cast as directed by you:

a. Six 1- by 1- by 10-in. steel mold prisms.

b. Three 3- by 6-in. steel cylinder molds.

c. Six 2-in. cube molds.

All of the above specimens were placed inside plastic bags, put in the fog room, and kept at 100 percent humidity and 73°F temperature immediately after casting. The cubes were demolded on Tuesday, March 27, and immediately returned to the fog room inside plastic bags. The prisms and cylinders were demolded April 3 and prepared for shipment.

I am enclosing a pint glass Mason jar that was filled at the same time the other specimens were cast on March 23. This jar was sealed and placed in the fog room together with the cast specimens. Daily observations were made each working day. The jar was not broken on Friday, March 30, but it was broken Monday, April 2. Therefore, expansion sufficient to break the glass jar occurred somewhere between 7 and 10 days after casting.

There was enough mixed grout to fill an additional eight 4- by 8-in. plastic cylinders plus one 6- by 12-in. plastic cylinder. These specimens were capped and are being kept at laboratory ambient temperature, about 73°F, in the open lab area. If you require additional specimens, these will be available to you upon request.

Particulars of the mixing operations on March 23, 1984, are:

a. Tap water was used.

b. Grout temperature was 69°F after mixing.

c. Flow was 24 seconds.

d. The water from the mix design sheet was used.

e. Mixing time before casting was about 30 minutes.

Let me know if I can be of further service.

Sincerely,

Donald M. Walley

Donald M. Walley
Concrete Technology Division
Structures Laboratory

APPENDIX D

29 August 1983 Grout Report

DEPARTMENT OF THE ARMY
WATERWAYS EXPERIMENT STATION, CORPS OF ENGINEERS
PO BOX 631
VICKSBURG MISSISSIPPI 39180

REPLY TO
ATTENTION OF

September 1, 1983

Structures Laboratory

Mr. D. P. Moak
Project Manager
Battelle Project Management Division, ONWI
505 King Avenue
Columbus, Ohio 43201

Dear Mr. Moak:

I am enclosing the information you requested on the grout we were to develop in a brief report (Enclosure).

As the report indicates, dry-batched samples of this grout and some extra cement will be sent to Dan Bush at Terra Tek this week.

Sincerely,

A. D. Buck

A. D. Buck
Materials and Concrete Analysis Group
Structures Laboratory

Enclosure

Corps of Engineers, USAE Waterways Experiment Station	Grout Report	Structures Laboratory P. O. Box 631 Vicksburg, Mississippi
Project Development of a Suitable Salt Grout for the 1983 Terra Tek Test		Date 29 August 1983 ADB, JAB, DW

Introduction

1. One of the FY 83 milestone requirements for the Office of Nuclear Waste Isolation (ONWI) project was to develop a grout considered suitable for the large-scale test to be conducted at Terra Tek in the Fall of 1983. Since that test involves casting grout against a salt specimen, there was general agreement that this would have to be a salt grout to prevent any dissolution of the salt specimen by water in the grout.

2. In addition, involved personnel in the Concrete Technology Division (CTD) of the Structures Laboratory (SL) felt that it would be best to use a sanded grout. This was based on several considerations; these included the fact that sanded grout would have less volume change than a nonsanded one in response to temperature and moisture changes; sanded grout would have less heat rise due to heat of hydration since the cement content is decreased. Finally, there was the repeated observation over a period of years that specimens made without sand tend to develop cracks during storage in the laboratory when there is some opportunity for temperature or moisture changes or both of these.

3. The nonsanded grout mixture BCT-1-F had been developed earlier for possible sealing application in salt but was never used. Since it did contain salt, it served as a starting point to develop a sanded salt grout.

Test procedure

4. Using the same or similar materials, several trial batches of modified BCT-1-F grout were made and evaluated for workability chiefly on the basis of flow. A promising mixture was made as an 0.3-ft^3 batch on 13 July 1983. Four bars were made for restrained expansion measurements in general accordance with ASTM Designation: C 806-75, "Standard Test Method for Restrained Expansion of Expansive Cement Mortar."(1) Two bars were 2 by 2 by 10 in. in size and the other two were 3 by 3 by 10 in. Six cubes, each 2 by 2 by 2 in., were made; two were broken at 5-days age, 1 at 7-days age, and 3 at 28 days to determine compressive strength. This was in general accordance with the requirements of ASTM C 109-80, "Standard Test Method for Compressive Strength of Hydraulic Cement Mortars."(1) One 6- by 12-in. cylinder was made for possible later testing.

5. When observation of the above cylinder showed that it had about 1 percent of bleed water on it, the mixture was changed again. This modification was made 15 August 1983 as an 0.1-ft^3 batch; nine cubes were made for strength testing as before.

(1) 1982 Annual Book of ASTM Standards, Part 13, American Society for Testing and Materials, Philadelphia, PA.

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Enclosure 1

Results

6. The mixture made on 15 August 1983 was considered best in that it had less bleeding than the 13 July mixture. The 15 August formulation is shown in Table 1 along with flow times over a 2-hr period, amount of bleeding, and actual unit weight. While no specific time of set data are shown, observation indicated final set was more than 2 days and less than 3 days. The change from the 13 July to 15 August mixture was removal of some silica flour and addition of more of the two sand sizes.

7. Two significant changes in the BCT-1-F grout were addition of sand and change to a different type of high-range water reducer (HRWR) (superplasticizer). The Materials Research Laboratory of the Pennsylvania State University (PSU) had found during 1983 that a melamine type of HRWR was more effective in preventing the usual high foam formation in salted mixtures than the naphthalene type (D-65) that had originally been used in BCT-1-F. After Waterways Experiment Station (WES) had confirmed this in small-scale concrete mixtures, it was decided to use the melamine type of HRWR.

8. Three separate 1-ft^3 dry batches of the mixture shown in Table 1 were sent to the attention of Mr. Dan Bush at Terra Tek the week of 29 August to 2 September 1983 for his use before and in the mid-September test.

9. Restrained expansion data for the 13 July mixture are shown in Table 2 through 47 days of testing. These data show expansion of 0.08 and 0.06 percent for the larger and smaller bars, respectively, at 47 days. While expansion was not measured for the 15 August mixture, it should be similar.

10. Compressive strength data for the mixtures cast 13 July and 15 August are shown in Table 3. The low average 3-day strength of 240 psi for the 15 August mixture is a reflection of the slow setting time associated with such salted mixtures. Its average strength was 3460 psi at 7 days and should be similar to the 5290-psi 28-day value shown for the 13 July mixture when cubes are tested 12 September 1983.

Conclusions

11. A grout mixture considered satisfactory for the September 1983 placement and October 1983 test at Terra Tek was developed. It contains enough salt to saturate the mixing water to prevent dissolution of the salt specimen when contact is made. It contains sand to minimize volume change effects. Plastic properties such as low flow times and minimal bleeding are considered good. Restrained expansion and compressive strength are considered satisfactory.

12. Setting times are between 2 to 3 days as compared to 1 day for nonsalted cement mixtures. These extended setting times have not been considered a problem. It is likely they could be shortened by use of an accelerator if this was desirable.

END

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